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MICROSCALE DISTILLATION

LIKE THE BIG GUY

Before you read this chapter, read up on Class 1: Simple Distillation (Chapter 20) so you'll know what I'm talking about.

Until recently, there was no way you could duplicate an ordinary distillation of any class in microscale (re-read the material on the different classes in Chapter 20, "Distillation"), because the equipment either didn't exist or was fabricated with glass joints that didn't match up. Recently, Ace Glass (and others for all I know) has produced an all $\text{T } 14/10$ joint microscale kit. Unfortunately neither a three-way nor a vacuum takeoff adapter is included. These pieces are, however, available separately.

So you *can* set up the same apparatus and do the same distillation for the same classes you've always done.

That's not completely true. Using this apparatus at the true microscale (0.01 mole), you have two drops of a liquid to distill. I'd see my instructor before I did any of these techniques with fewer than 10 ml.

Class 1: Simple Distillation

Perhaps you'll be using a conical vial rather than an R.B. flask, but the setup and operation are the same as with the larger apparatus. **Note:** the tubing carrying cooling water can be so unwieldy that moving the tubing can pull this tiny setup out of alignment.

Class 2: Vacuum Distillation

Because the setup is so small and your sample size is so small, you've got to be very careful here. With $\text{T } 14/20$ ware (it's a little bigger), the operations are identical to those of the $\text{T } 24/40$ or $\text{T } 19/22$ sizes. I don't *have* the $\text{T } 14/10$ setup yet, so I can only extrapolate. You'll have the same entertaining experiences. Watch the vacuum hose, too. It can be so unwieldy that it pulls the apparatus out of line if it's moved.

Oh. Check out the "O-ring cap seal" discussion for a perspective on microscale vacuum distillation (see Chapter 6, "Microscale Jointware").

Class 3: Fractional Distillation

You'll probably substitute one of those **air condensers** for the distilling column. As a straight, unjacketed tube, the air condenser is not very efficient. I'd be careful about packing it with anything; the column holdup, as small as it is, is likely to hold up a lot of your product.

Class 4: Steam Distillation

With sample sizes this small, internal steam distillation is the only way out. With the microscale equipment, you can inject water by syringe rather than opening the apparatus up to pour water in, and lose less product. Again, if your sample is very small, see your instructor about any problems.

MICROSCALE DISTILLATION II: THE HICKMAN STILL

If you have only 1–2 ml to distill, then the Hickman still is probably your only way out (Fig. 107).

The Hickman Still Setup

1. Set up the stirring hot plate and sand bath. I suppose you *could* stick a thermometer in the sand to give you an idea of how warm the sand is.
2. Put the sample you want to distill into an appropriately sized conical vial. Two things: Don't fill the vial more than halfway, and don't use such a big vial that the product disappears. Less than one third full, and you'll lose a lot on the walls of the vial.
3. These vials can fall over easily. Keep them in a holder, or clamp them until you're ready to use them.
4. Add a microboiling stone *or* a spin vane, not both, to the vial.
5. Get a clamp ready to accept the Hickman still at about the position you want; just open and loosely clamped—horizontally—to the ringstand.
6. Scrunch the conical vial down into the sand, and push it to where you want it to be. Hold the vial at the top.

7. Put the Hickman still on the vial. Now, even though you have a sand buttress, this baby is still top heavy and can fall. Hold the vial **and** the still somewhere about where they meet.
8. Jockey the clamp and/or the vial and still so that they meet. Clamp the still at the top in the clamp jaws. Get the vial and still in their final resting place; tighten the clamp to the ringstand.
9. Hang a thermometer by a clamp and stopper so that the thermometer goes straight down the throat of the still. Place the thermometer bulb at about the joint of the vial and still. Do *not* let the thermometer touch the sides of the vial or still.
10. You're ready to heat.

Hickman Still Heating

You don't have very much liquid here, so it doesn't take much heat to boil your sample. If you have no idea at what temperature your sample will boil at, you should just heat carefully until you see it boil, and then *immediately* turn the heat down 10–20%. Of course, you have a microboiling stone or spinning spin vane in there, don't you?

As you heat the sample, you'll see a ring of condensate travel up the vial, and when it hits the thermometer bulb, the temperature reading should move up. As usual, if there's **refluxing liquid** on the bulb and the temperature has stabilized, that's the boiling point.

The vapor then travels up into the still head, condenses on the glass surface, and falls down, to be caught in the bulge of the still (Fig. 107).

When you feel you've collected enough liquid (this will depend on the experiment), you can go after it with a pipet and rubber bulb.

Recovering Your Product

If you have a thermometer down the throat of the **Hickman still**, forget about getting your product out with a disposable pipet; there's just not enough room, period. If you use a 5- $\frac{3}{4}$ inch pipet, you'll likely snap the tip off in the still when you try to bend it into reaching for your product, caught in the bulge of the still. Yes, you *can* just about wangle the end of a 9 inch pipet into the crevice and pick up your product, but there's a good chance

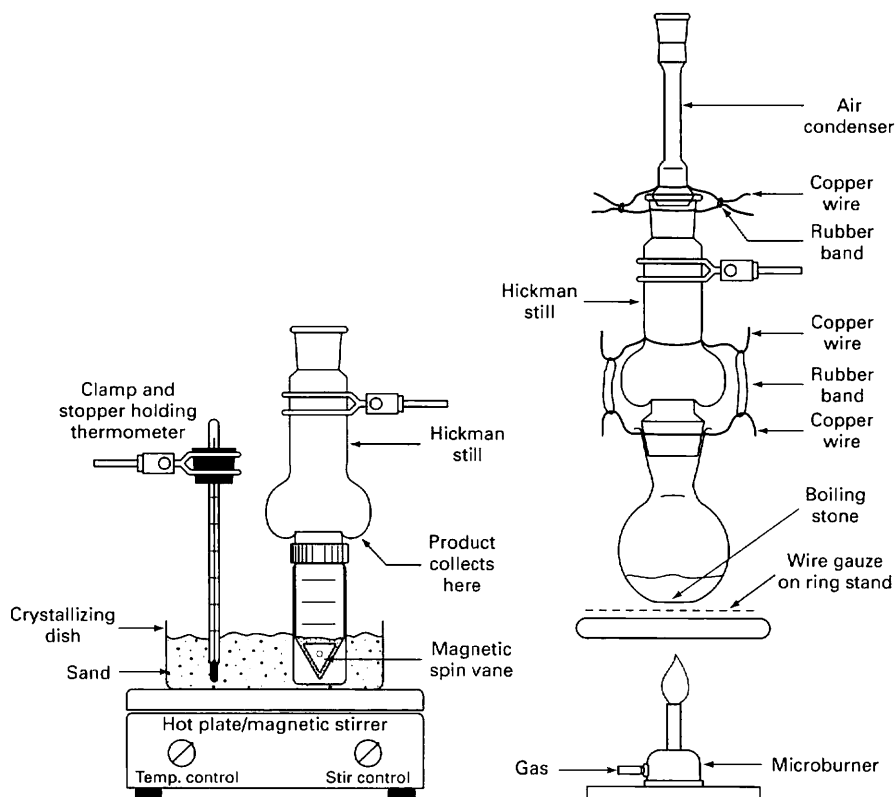


Fig. 107 Two different Hickman Still setups.

of breaking the tip off anyway. You might wind up having to put a bend in the tip, using a burner and your considerable glassworking skills. You have to use a small burner, and a small flame, and heat the tube carefully so it doesn't close off or melt off—in short, see your instructor about this. You should wind up with a slightly bent tip at the end of the 9 inch pipet. What I haven't yet tried, and wonder why it hasn't been done yet, is to put a small length of the gas-collecting tubing—very thin, narrow plastic or Teflon tubing—on the end of a pipet bent in a curve so you can go after your distillation product without resorting to glassworking. Again, the 9 inch pipet *can* just make it, but be very careful.